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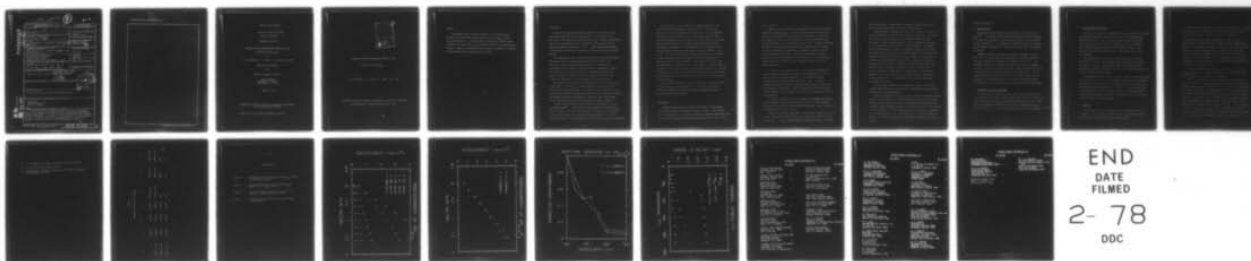
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PREPARATION AND PHOTOELECTROLYTIC BEHAVIOR OF THE SYSTEMS WO(3---ETC(U)
NOV 77 C E DERRINGTON, W S GODEK, C A CASTRO N00014-77-C-0387

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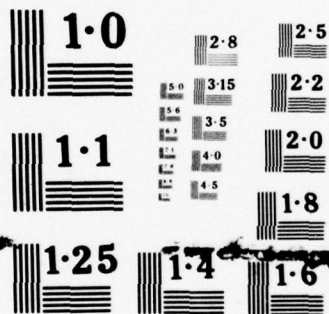
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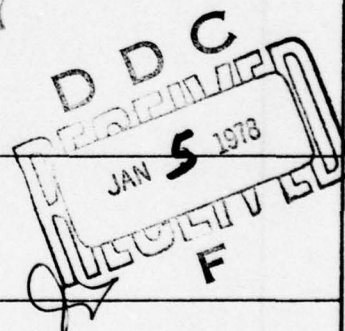
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WO_{3-x}WO_{3-x}F_x

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TECHNICAL REPORT NO. 2

PREPARATION AND PHOTOELECTROLYTIC BEHAVIOR OF THE
SYSTEMS WO_{3-x} AND $\text{WO}_{3-x}\text{F}_x$

by

C. E. Derrington, W. S. Godek, C. A. Castro, and A. Wold

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PREPARATION AND PHOTOELECTROLYTIC BEHAVIOR OF THE

SYSTEMS WO_{3-x} AND $WO_{3-x}F_x$

C. E. Derrington, W. S. Godek, C. A. Castro, and A. Wold

Contribution from the Department of Chemistry and Division of Engineering
Brown University, Providence, Rhode Island 02912

ABSTRACT

A convenient method of preparing compounds in the system $\text{WO}_{3-x}\text{F}_x$ is described and the photoelectrolytic behavior of WO_{3-x} and $\text{WO}_{3-x}\text{F}_x$ anodes has been investigated. It was found that the substitution of small amounts of fluorine for oxygen in WO_3 does not affect adversely its photoelectrolytic behavior. Moreover, substituted fluorine increases the stability of electrodes of WO_3 when used for the photoelectrolysis of water.

INTRODUCTION

There has been considerable interest recently in the search for stable electrode materials for use in photoelectrolysis. This process is one in which water is decomposed into hydrogen and oxygen when a suitable semiconductor, placed in an electrochemical cell, is illuminated with energy greater than the band gap, creating electron-hole pairs:¹ Under appropriate conditions, these electron-hole pairs can be separated and used as oxidizing and reducing species.²

The requirements of such a stable electrode material are indeed stringent. The material must be chemically inert and should have a band gap that utilizes an optimum of the solar spectrum (~ 1.6 eV). In addition, the semiconductor must be conducting and when placed in an electrolyte it should have sufficient band bending to separate the photogenerated electron-hole pairs. The position of bands relative to the electrochemical scale must be such that oxidation of O^{2-}/O_2 and reduction of H^+/H_2 can take place.

The degree of band bending and the position of the bands can be controlled to some degree by varying the pH of the electrolyte used in the cell and by the application of an external bias. It is known that the conductivity of such materials can be altered by chemical substitution or doping procedures. Therefore, the properties which are most difficult to control and, thus, more critical are the magnitude of the band gap and the material's chemical stability.

To date, the only materials which have been used successfully have been broad band oxides.³⁻⁶ These oxides were made conductive by removing small amounts of oxygen. Since there is some question as to the long term stability of these defect structures in a highly oxidizing environment⁷ it was of interest to compare the relative stability of defect oxides to those of oxyfluorides.

The use of WO_3 as a stable electrode in photoelectrolysis has been reported by several investigators. Butler, et. al.⁸ have indicated that WO_3 is an n-type semiconductor with a band gap of 2.7 eV. Experiments performed with single crystals in a 1.0M sodium acetate solution showed spontaneous photocurrent (i.e. no applied bias) with no decay in photocurrent or visible deterioration of the electrode surface even after passage of approximately 75 coulombs/cm². They indicated that an appreciable applied bias was necessary before photodecomposition of water was observed. Hodes, et. al.⁹ found similar results with polycrystalline films of WO_3 prepared by either the oxidation of tungsten metal films or the decomposition of ammonium tungstate. Hardee and Bard¹⁰ have prepared WO_3 films by three different methods. Unlike Hodes, et. al, they found that the films prepared by the oxidation of the metal were not stable on repeated cycling of the electrode. Unfortunately, the electrode surfaces were deep blue rather than yellow-green. This color difference is indicative of either reduced WO_3 or hydrogen bronzes of the type H_xWO_3 .

It was therefore desirable to examine more closely the photoelectrolytic behavior of WO_{3-x} for various amounts of x. It was also of interest to determine the effect on stability of substituting fluorine for oxygen in WO_3 , rather than creating defects.

EXPERIMENTAL

Tungsten oxide was prepared by heating high purity (99.99%) tungsten foil (0.010") under flowing oxygen for 24 hours at 1000°C. Defect tungsten oxide films were made by heating the WO_3 samples in evacuated sealed silica tubes in the presence of freshly ground titanium at temperatures of 100°C to 800°C for

2 to 24 hours.¹¹

A convenient method for substituting fluorine has been used to synthesize members of the system $WO_{3-x}F_x$. In this process the freshly oxidized WO_3 was placed on a pure tungsten foil which sits in a tungsten boat. The boat was then placed in one zone of a two zone furnace. It was determined that the optimum temperature of this reaction zone should be 650°C. Potassium bifluoride was placed in a nickel boat and inserted in the second zone of the furnace. This zone was maintained at 400°C which was sufficient to allow for a slow thermal decomposition of the KHF_2 . Variation in the fluorine content was achieved by varying the temperature of the reaction zone. The entire reaction chamber consisted of a nickel tube with stainless steel Swagelok fittings welded to both ends.

The reaction tube was purged initially with argon at room temperature. After 30 minutes the gas flow was stopped and the temperature of the two zones was raised to operating conditions. It was found that uniform samples were made under these static conditions. After 4 hours the reaction tube was removed from the furnace and flushed again with argon until room temperature was achieved. During the purging procedures the argon was bubbled through a 9M NaOH solution. All reactions were carried out in a well ventilated hood.

The crystal structures of all of the samples were determined from powder x-ray diffraction spectra using either MgO or CaO as an internal standard. The scans were taken with a Philips Morelco Diffractometer, using $CuK\alpha$ radiation (1.5405\AA) at a rate of $1/4^\circ 20 \text{ min}^{-1}$.

Resistivity measurements were made by ultrasonically soldering indium leads on the polycrystalline samples and using the standard four-probe van der Pauw technique.¹² Optical band gap measurements were made by making KBr windows

which contained finely ground oxide or oxyfluoride intimately mixed and the absorption spectra were taken on a Cary 14 spectrophotometer.

Photoelectrolytic measurements were made by first soldering an oxide or oxyfluoride sample to a Pt wire which was sealed in a pyrex tube. All but the face of the film was then coated with an electrically insulating resin (Microstop, Michigan Chrome and Chem. Co.). The electrode was then placed in a silica cell which was filled with 0.2M sodium acetate. A platinized Pt foil with approximately 5 times the geometrical surface area of the oxide and oxyfluoride films was used as the counter electrode. The electrodes were illuminated by either the full output of a 150-W Xenon lamp or the output of a Model 7155 monochromator (Oriel Corporation). Absolute light intensity measurements were made using a Pyroelectric Radiometer (Model RK 3440, Laser Precision Corp.). The current vs wavelength curves were corrected for intensity variations by normalizing the spectral output of the lamp-monochromator system to 440 nm and using the resulting values to correct the measured photocurrents. All measurements were made with the electrolyte in equilibrium with air.

Stability against reoxidation was determined by heating the films in a stream of flowing oxygen at a rate of 100°C/hr to 1000°C and the weight change monitored with a Cahn Electrobalance.

Analysis for fluorine in the $\text{WO}_{3-x}\text{F}_x$ samples was made by dissolving approximately 0.2g of the samples in 9 M NaOH, adjusting the pH to 5.5 with a sodium acetate/acetic acid buffer and measuring the fluoride ion concentration using a fluorine specific electrode (Model 94-09, Orion Research, Inc.). By the use of a specific electrode, F^- ion concentrations of 24 $\mu\text{g./liter}$ could be determined with a reproducibility of $\pm 2\%$. From the weight of the samples analyzed, the value of x in the $\text{WO}_{3-x}\text{F}_x$ samples can be reported with an accuracy equivalent to ± 0.0002 .

RESULTS AND DISCUSSION

1. Crystallography

There have been conflicting reports concerning the structure of pure WO_3 .¹³⁻¹⁶ It was found that WO_3 prepared by oxidizing completely tungsten foil could best be indexed on a triclinic system similar to the one reported by Roth and Waring.¹⁶ It transforms to a monoclinic phase upon the removal of small amounts of oxygen¹⁷ or the addition of small amounts of hydrogen or sodium.¹⁸

Whereas the system WO_{3-x} ($0 < x \leq 1$) is monoclinic throughout the entire range,¹⁷ the system $\text{WO}_{3-x}\text{F}_x$ ($0 < x \leq 1$) undergoes the following transitions: triclinic \rightarrow monoclinic \rightarrow orthorhombic \rightarrow tetragonal \rightarrow cubic.^{19,20} The structural properties of the $\text{WO}_{3-x}\text{F}_x$ prepared in this study are summarized in Table I. It can be seen that the structure remains triclinic for very small ($x=0.0079$) amounts of substituted fluorine in the system $\text{WO}_{3-x}\text{F}_x$. When the amount of substituted fluorine is $x=0.0177$ the structure has undergone the transition to the monoclinic phase and when $x=0.0663$ the resulting phase is orthorhombic. These are consistent with results of previous investigators.

2. Resistivity and Optical Properties

The resistivity of the pure WO_3 samples were between 10^6 and $10^8 \Omega\text{-cm}$. The resistivities of the WO_{3-x} samples ranged between $1.2 \times 10^4 \Omega\text{-cm}$ - $7 \times 10^{-1} \Omega\text{-cm}$ and the resistivities of the $\text{WO}_{3-x}\text{F}_x$ samples ranged between $100 \Omega\text{-cm}$ - $5 \Omega\text{-cm}$. Absorption measurements indicated that the band gap of WO_3 and all $\text{WO}_{3-x}\text{F}_x$ samples was $2.65 \pm 0.10 \text{ eV}$. This is consistent with previous investigations.^{8,9,21,22}

3. Photoelectrolytic Properties

In Figure 1 are plotted the photocurrents vs applied voltage (SCE reference) for several WO_{3-x} samples. Measurements were made with the electrolyte in equilibrium with air. As can be seen the largest photocurrent is reached for the WO_{3-x} sample having the lowest resistance, with the photocurrents of the remaining samples decreasing as the resistance increases. These results are consistent with the only effect being a change in the overall cell resistance.

The photocurrents for two triclinic samples of $\text{WO}_{3-x}\text{F}_x$ are shown vs. applied bias in Figure 2, the measurements being made with the electrolyte in equilibrium with air. Although photocurrents were observed for all of the tungsten oxyfluorides studied, the monoclinic and orthorhombic compositions ($x \geq 0.0177$) show more complex behavior.

The spectral response of the $\text{WO}_{3-x}\text{F}_x$ samples shown in Figure 3 were obtained with an applied bias of 0.5V. The photocurrents plotted here were normalized for clarity by taking the ratio of the photocurrent at a given wavelength to the maximum photocurrent obtained (i.e. at 400 nm). The actual photocurrents at 400 nm are for $x = 0.0079$ $I = 16.06 \mu\text{A}/\text{cm}^2$ and for $x = 0.0083$ $I = 6.07 \mu\text{A}/\text{cm}^2$. The colors of the materials varied from a light green for the $x = 0.0079$ sample to darker green for the $x = 0.0083$ sample.

4. Stability

Stability of the WO_{3-x} and $\text{WO}_{3-x}\text{F}_x$ samples was investigated by three procedures: Stability against reoxidation, stability against hydrolysis or dissolution, and stability in a working cell arrangement.

Figure 4 shows the results of the reoxidation experiments. It can be seen that while WO_{3-x} readily reoxidizes at 250-300°C, the $WO_{3-x}F_x$ sample was stable to 600°C, implying an increased stability to reoxidation.

It is also clear that the thermal gravimetric data shown in Figure 4 indicates that the composition of the oxyfluoride samples prepared in this study can be represented by the formula $WO_{3-x}F_x$. If the composition of the samples was of the stoichiometry $WO_{3-x}F_y$, then there would have been a gain in weight recorded equivalent to the value of $x-y$. It can be seen from Figure 4 that the sensitivity of this method is such that values of x (representing an oxygen deficiency) in the system WO_{3-x} can be determined where values of x are less than 0.01.

Hydrolysis and dissolution experiments were made by placing samples of WO_{3-x} and $WO_{3-x}F_x$ in 0.2M H_2SO_4 and leaving these at 90°C for 350 hours. Neither sample showed any hydrolysis or dissolution.

Stability in a working cell, with the electrolyte in equilibrium with air, was determined by biasing the electrode at 0.5 volts with respect to the platinum cathode, illuminating then with the full output of a 150 watt Xenon lamp and monitoring the changes in photocurrent with time. It was found that while the slightly reduced WO_{3-x} films gave stable photocurrents, the more reduced samples were less stable. The most reduced sample ($x \approx 0.03$) was very unstable with the photocurrent decreasing by 30% over a period of 2 hours. This is consistent with the results found by Hardee and Bard.¹⁰ On the other hand, the triclinic samples of $WO_{3-x}F_x$ gave stable photocurrents of 2ma/cm^2 for periods up to 46 hours (~ 700 coulombs). In addition, there was no significant weight loss and no visible change on the surface of the electrodes.

CONCLUSIONS

The photoelectrolytic behavior of WO_{3-x} and $\text{WO}_{3-x}\text{F}_x$ has been investigated. It was found that substituting fluorine for oxygen in WO_3 , in small amounts, does not affect adversely the photoelectrolytic behavior of WO_3 but increases the stability of the compound when used as an electrode for photoelectrolysis of water. A safe and easily controlled method of preparing $\text{WO}_{3-x}\text{F}_x$ was described.

ACKNOWLEDGEMENT

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TABLE 1

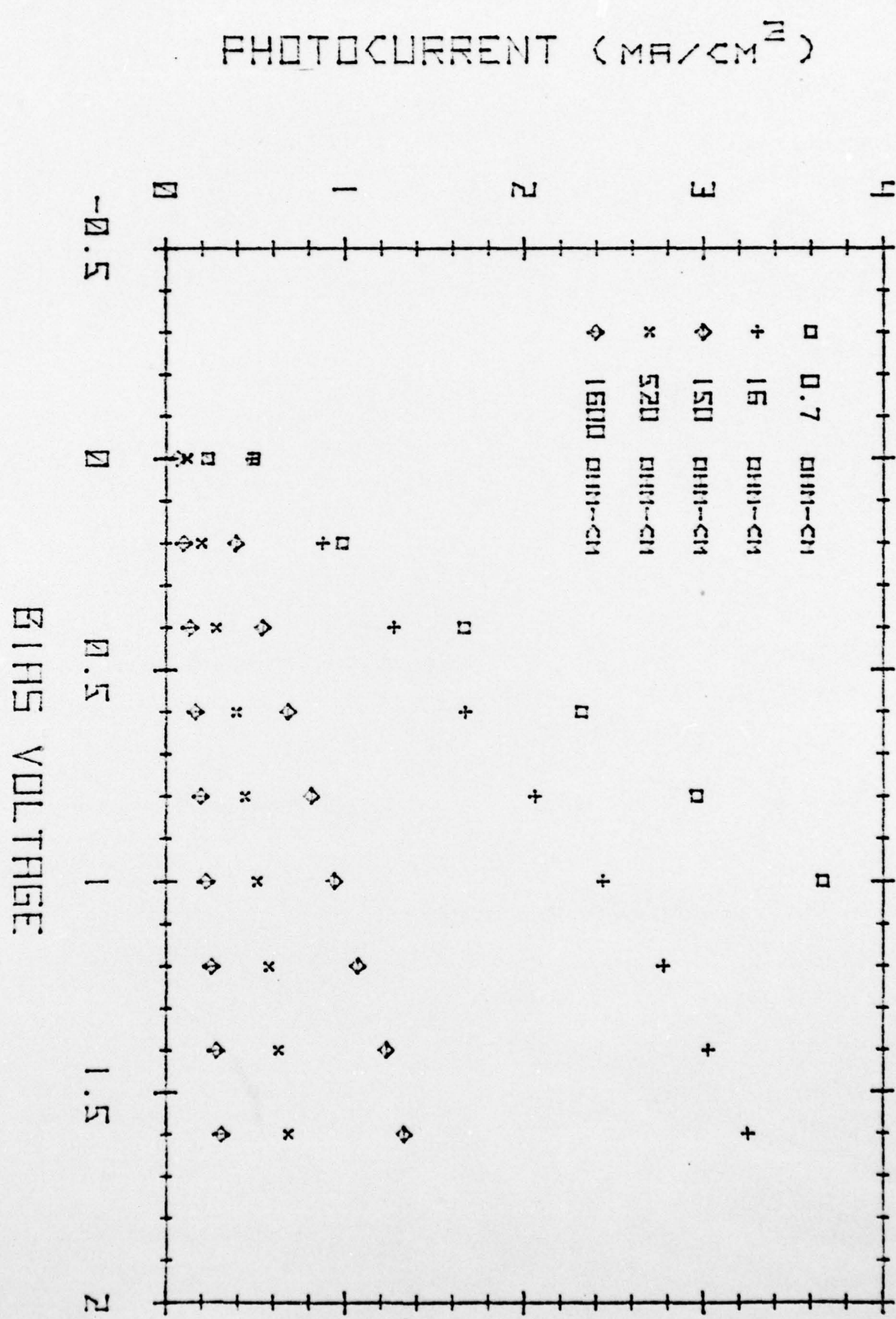
Structural Properties of $\text{WO}_{3-x}\text{F}_x$

x	a	b	c	α	β	δ
0	7.306(1)	7.527(1)	3.854(1)	$88^\circ 43' \pm 02'$	$90^\circ 17' \pm 02'$	$90^\circ 39' \pm 02'$
0.0079 ± 0.0005	7.301(1)	7.527(1)	3.856(1)	$88^\circ 48' \pm 0^\circ 02'$	$90^\circ 17' \pm 02'$	$90^\circ 35' \pm 02'$
0.0177 ± 0.001	7.311(1)	7.545(1)	3.851(1)	90°	$90^\circ 51'$	90°
0.0663 ± 0.005	7.369(1)	7.482(1)	3.848(1)	90°	90°	90°

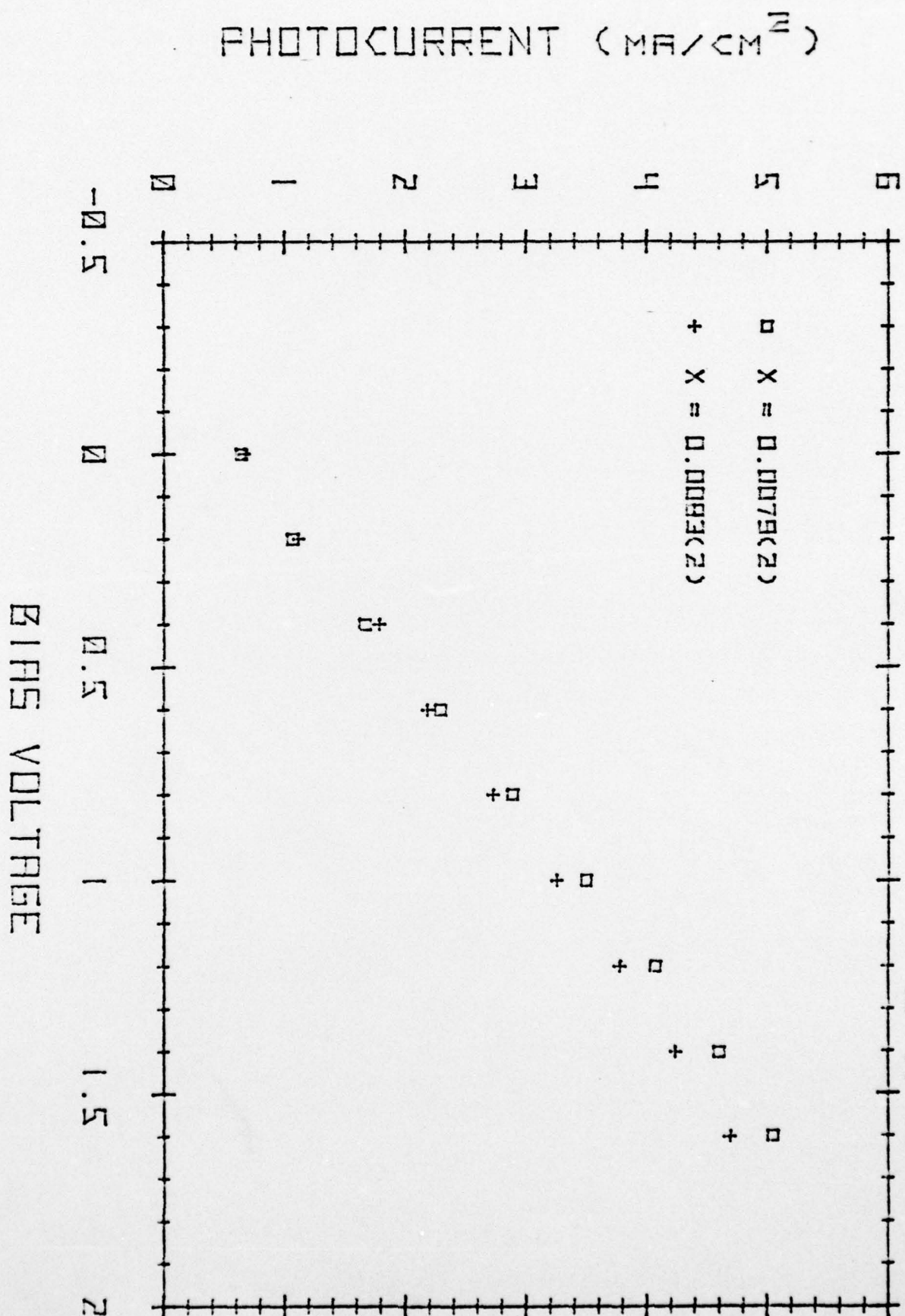
FIGURE CAPTIONS

- Figure 1 - Photocurrent vs applied bias for several WO_{3-x} samples in 0.2M $\text{NaC}_2\text{H}_3\text{O}_2$ (pH=7.8).
- Figure 2 - Photocurrent vs applied bias for several $\text{WO}_{3-x}\text{F}_x$ samples in 0.2M $\text{NaC}_2\text{H}_3\text{O}_2$ (pH=7.8).
- Figure 3 - Normalized Spectral Response of $\text{WO}_{3-x}\text{F}_x$ in 0.2M $\text{NaC}_2\text{H}_3\text{O}_2$. E_g =optical band gap (2.7 eV).
- Figure 4 - Stability against reoxidation for WO_{3-x} and $\text{WO}_{3-x}\text{F}_x$ in flowing oxygen.

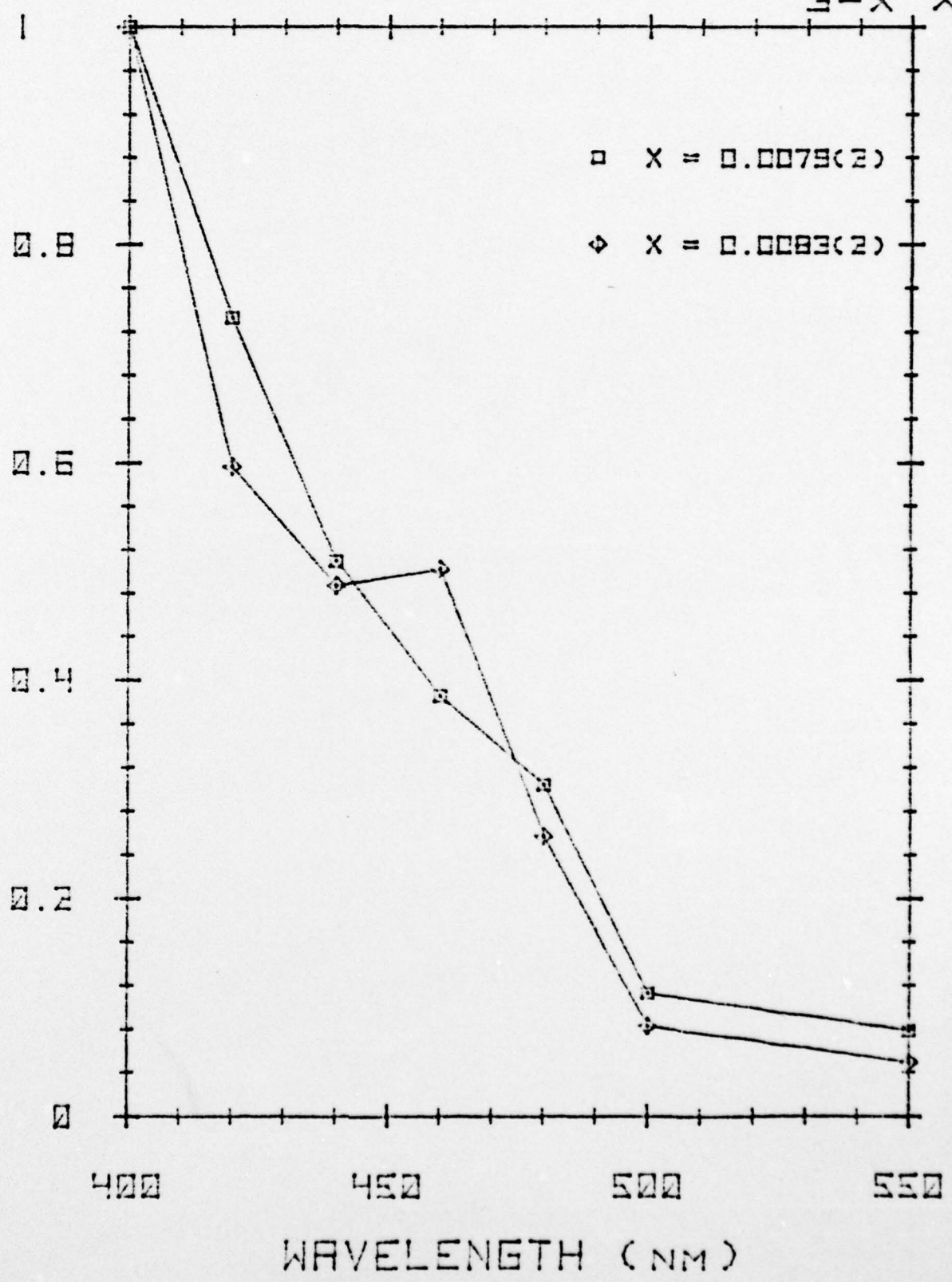
PHOTOCURRENT OF WD_{E-X}



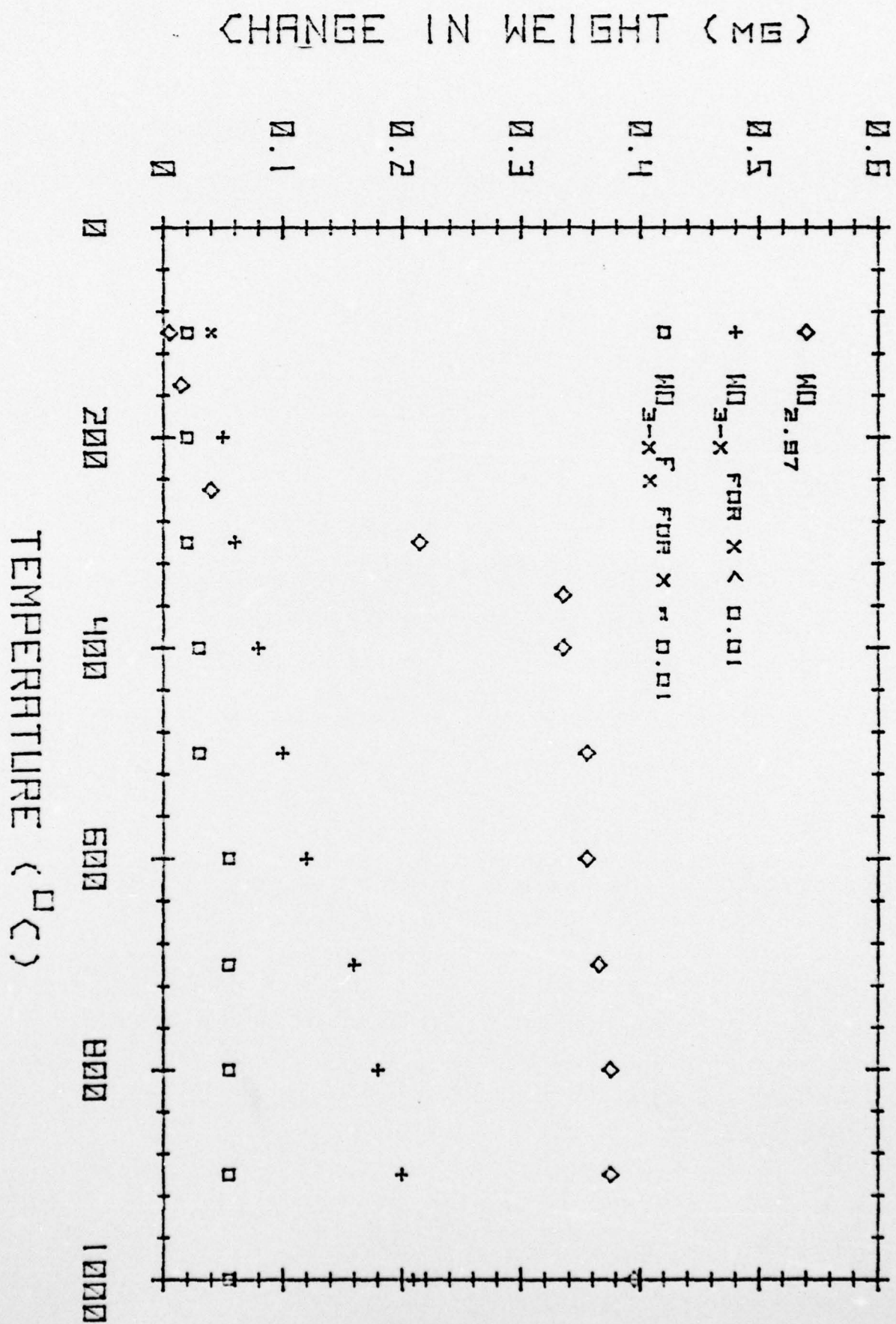
PHOTOCURRENT OF $\text{WO}_{3-x}\text{F}_x$



NORMALIZED PHOTOCURRENT

SPECTRAL RESPONSE OF $\text{WO}_3\text{-X}\text{F}_\text{x}$ 

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